

Description

A Method Of Establishing Stress Relieving Procedures For Minimizing Sulfide Stress Cracking In Cold Worked Metals

BACKGROUND OF INVENTION

[0001] Field of the Invention.

[0002] The present invention relates generally to methods for establishing effective heat-treating processes and the manufacturing of products using such heat-treating process. More specifically, the present invention relates to a method for establishing optimum stress relieving procedures for reducing the susceptibility to SSC in cold worked metals and to the metal products made from materials processed by such optimized procedures.

[0003] Background Setting of the Invention.

[0004] Cold working metals may cause the metal to exhibit undesired physical characteristics. A common effect of cold

working is an increase in hardness of the material and a decrease in the ductility of the material. Cold working may also cause a decrease in the resistance of a material to the effects of corrosion and other chemical reactions.

- [0005] The effects of cold working in the steel pipe used in the construction and production of oil and gas wells and in other chemical applications are well known. Swaging or cold working steel pipe ends, or any steel product for that matter, will cause an increase in hardness, a decrease in ductility and a decrease in the resistance of the steel to the effects of sulfide stress cracking (SSC) in hydrogen sulfide (sour) environments. The presence of hydrogen sulfide in oil and gas wells dictates that preventive measures be taken to prevent SSC in the tubulars used in the construction and production of such wells.
- [0006] The National Association of Corrosion Engineers (NACE) recognizes that cold working causing a change in dimensions of the material by over 5% can be detrimental to service in hydrogen sulfide (sour) environments. NACE Standard MR0175 addresses a minimum recommended temperature for stress relieving cold worked materials. The Standard provides that if tubulars and other components are cold straightened at or below 950 degrees

Fahrenheit, they shall be stressed relieved at a minimum of 900 degrees Fahrenheit. The Standard also specifies that if the tubulars and tubular components are cold formed (pin nosed and/or box expanded) and the resultant permanent outer fiber deformation is greater than 5%, the cold-formed regions shall be thermally stress relieved at a minimum temperature of 1100 degrees Fahrenheit. Cold forming the connections of high strength tubulars with hardnesses above 22 HRC requires thermal stress relieving at a minimum temperature of 1100 degrees Fahrenheit.

- [0007] Swaging or cold working of steel pipe bodies is a common procedure in the manufacture of certain tubular goods. It has been determined that stress relieving of cold worked materials at temperatures less than 1100 degrees Fahrenheit (the NACE MR0175 minimum) is effective in lowering the resistance of such materials to SSC. It has also been determined that tempering the material at higher temperatures does not necessarily return the materials SSC resistance to its original corrosion resistance prior to swaging and stress relieving. Testing for evaluating the effectiveness of tempering to improve SSC resistance has usually included either the NACE standardized test method

TMO177–Double Cantilever Beam Test Method D or the less revealing NACE test TMO177–Method A–Tension Test. The Method A test is a quantitative pass/fail type test, and as such does not accurately depict the deterioration in SSC resistance due to cold working. It also does not reflect the effectiveness of the stress relieving process in returning the material to its original condition of SSC resistance prior to cold working.

- [0008] The use of induction heating for tempering or stress relieving cold worked pipe ends has been found to be inadequate to eliminate degradation in SSC resistance. Traditional temperatures and duration of heating in induction process can be grossly inadequate. Determining the proper combination of temperature and time exposures for a given material composition to reestablish a SSC resistance in the treated material equivalent to that existing before the material has been cold worked is a difficult and time-consuming process.
- [0009] Because the Method A NACE tests is a pass/fail type, one may only deduce that the specific time and temperature applied to a test sample during the stress relieving process were adequate to achieve a desired SSC resistance. Even if the treated sample passes the SSC resistance test,

the temperature employed in the stress relief process may have been higher than necessary and the time of exposure may also have been longer than necessary. Moreover, the time required to perform either of the NACE tests is extremely long, a factor, which must be considered in the production process. Passing the Method D test requires a 14-day process. Passing the Method A test requires a 30-day process. First discovering that a stress reduction process has failed to provide a desired SSC resistance as much as 30 days after the stress relieving process has occurred can pose serious manufacturing concerns.

- [0010] Plastic deformation associated with cold working produces an increase in the number of dislocations in the material within a given area. Metal that has been cold worked takes on a condition of higher internal energy than that of undeformed metal. The cell structure of cold worked metal is mechanically stable and thermodynamically unstable. The stability of the cold worked material becomes less stable with increasing temperatures. With sufficient heat and time of exposure, the metal softens and will revert to a stress-free condition. During the process of reverting to its earlier stress-free condition, the material of the metal goes through a recrystallization phase with accompanying

grain growth. The metal is said to have "recovered" when its physical properties are restored without any observable change in microstructure. The overall process by which this recovery occurs is known as stress relieving.

- [0011] The prior art is able to measure various properties of metals with procedures that provide immediate results. It is known, for example, that the electrical conductivity of a heat-treated, cold worked material increases rapidly toward the stress relieved value during recovery, and lattice strain, measured with X-rays, becomes appreciably reduced. Recrystallization of the metal may be readily detected by metallographic methods as is evidenced by a decrease in hardness or strength and an increase in ductility. The density of dislocations also decreases considerably on recrystallization and all the effects of strain hardening are eliminated.
- [0012] Various other techniques exist for evaluating the characteristics of metals without resorting to testing procedures that require extended time periods to complete. By way of example, the evaluation of dislocation densities to obtain information about the hardness of the material is an accepted procedure that produces information quickly and accurately. The dislocation density of a cold worked metal

and the use of the thin-film electron microscope in determining the dislocation density are well described in Mechanical Metallurgy, Third Edition by George E. Dieter, page 230, noting:

[0013] "Considerable detail knowledge on the structure of the cold-worked state has been obtained from thin-film electron microscopy. In the early stages of plastic deformation slip is essentially on primary glide planes and the dislocations form coplanar arrays. As deformation proceeds, cross slip takes place and multiplication processes operate. The cold-worked structure forms high-dislocation-density regions or tangles, which soon develop into tangled networks. Thus, the characteristic structure of the cold-worked state is a cellular structure in which high-density-dislocation tangles form the cell walls. The cell structure is usually well developed at strains of around 10%. The cell size decreases with strain at low deformation but soon reaches a fixed size, indicating that as strain proceeds the dislocations sweep across the cells and join the tangle in the cell walls. The exact nature of the cold-worked structure will depend on the material, the strain, the strain rate and the temperature of deformation".

[0014] While techniques exist for rapid, and accurate methods for evaluating some characteristics of treated or processed metals, the prior art has not proposed a speedy, quantitative procedure that permits establishing a stress relieving process for returning a cold worked metal to its original SSC resistance. The problem is complicated by the fact that stress-relieving procedures for restoring a cold worked material's resistance to SSC vary with the composition and configuration of the material to be stress relieved.

[0015] Following established heat-treating procedures for stress relieving cold worked metals does not assure that a given stress relieved material will exhibit adequate SSC resistance. It is well known, for example, that if the new strain-free grains resulting during a stress relieving process are heated at a temperature greater than that required to cause recrystallization, there will be a progressive increase in grain size. Treating a particular material to achieve desired results requires precise applications of heating and times of exposure. For these reasons, as well as others, the prior art has lacked a procedure for making an early determination that a heat-treating process has been effective in restoring the SSC resistance of a cold

worked material. Testing procedures that can take up to 30 days before confirming that a treated material has adequate SSC resistance make the availability of appropriate heat-treating processes essential to efficient manufacturing practice.

SUMMARY OF INVENTION

- [0016] The present invention uses the correlation between dislocation density in an stress relieved (tempered) metal having a known SSC resistance before cold working and the dislocation density of the metal after cold working and stress relieving for predicting the SSC resistance of a cold worked material following stress relieving.
- [0017] In another aspect of the present invention, the dislocation density of a cold worked metal that has been stress relieved is evaluated to determine the temperature and time of exposure requirements for a stress relieving process that will restore a SSC resistance level in the stress relieved material approximating that existing before the material is cold worked.

DETAILED DESCRIPTION

- [0018] The effectiveness of a selected combination of temperature and time exposures during a stress relieving process

for a specific material may be evaluated by examining the dislocation density of the treated material.

- [0019] The results of the evaluation may be used to determine an optimum temperature/time relationship for stress relief to achieve a desired SSCresistance. The dislocation density results for the determined optimum temperature/time relationship can be verified using conventional NACE testing. The evaluation of the dislocation density provides a predictive basis for anticipating compliance of a specific temperature/time relationship for a given metal specimen with the NACE tests. Advance knowledge that the heat-treated specimen will pass the NACE test avoids the necessity for retreating the material and enduring a second extended delay for subsequent NACE testing.
- [0020] The dislocation density of a specimen is preferably evaluated with the use of a transmission electron microscope (TEM). Within the pertinent ranges of temperature and time of during the stress relief treatment, the dislocation density of the metal decreases as the temperature and time of heating are increased. Use of the dislocation density of the sample as an indicator of SSC resistance permits immediate prediction of compliance of the sample with the applicable NACE standards. The prolonged test-

ing period required by the NACE procedures to confirm SSC resistance may thus be limited to a single test.

[0021] The dislocation density of a selected material may be determined with a transmission electron microscope prior to cold working to establish a baseline for subsequent comparison. Following the cold working and heat treating procedure, the dislocation density of the selected material may be again determined with the transmission electron microscope to compare it with the base line established for the material before the initial cold working. The heat treating process for the cold worked material may be varied and the dislocation density of the resulting heat treated material may be evaluated to determine the temperature and time exposure required to best bond be achieve a dislocation density most closely approximating that of the material before the cold working.